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(54) FRAME SEALING AGENT COMPOSITION FOR LIQUID CRYSTAL DISPLAY

(57)Abstract:

PURPOSE: To provide the subject composition capable of improving the workability in producing simply in a short time high-grade liquid crystal displays.

CONSTITUTION: The objective curable composition essentially comprising (A) at least one kind of partially (meth)acrylated epoxy resin obtained by reaction of a bisphenol A-type epoxy resin with (meth)acrylic acid, (B) (meth)acrylic ester monomer(s) or oligomer (s) thereof, (C) an epoxy resin, (D) a photopolymerization initiator, and (E) a potential epoxy curing agent. The water-soluble ionic substance content of this composition is $\leq 40 \mu\text{S/cm}$ in terms of ionic conductivity.

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CLAIMS

[Claim(s)]

[Claim 1] a) The frame sealing-compound constituent of the liquid crystal display characterized by being the hardenability compound which uses a kind, b acrylic ester, methacrylic ester monomers or these oligomer, c epoxy resin, d photopolymerization initiator, and e latency epoxy curing agent as an indispensable component at least among the formation of a partial acrylic reacted and obtained in the bisphenol A mold epoxy resin, an acrylic acid, or a methacrylic acid, or a partial methacrylic-ized epoxy resin, and the content of the water meltable ionicity matter being 40 or less second/cm in whenever [ionic conduction].

[Claim 2] The frame sealing-compound constituent of the liquid crystal display of claim 1 whose content of the water meltable ionicity matter of claim 1 is 15 or less second/cm in whenever [ionic conduction].

[Claim 3] The frame sealing-compound constituent of the liquid crystal display of claim 1 which comes to blend f inorganic bulking agent with the hardenability compound of claim 1 further.

[Claim 4] The loadings of the acrylic ester which is the component b of claim 1, methacrylic ester monomers, or these oligomer are at least one sort in the formation of a partial acrylic which is Component a, or a partial methacrylic-ized epoxy resin. Frame sealing-compound constituent of the liquid crystal display of claim 1 which is 20 - 80 weight section to the 100 weight sections.

[Claim 5] The loadings of the epoxy resin which is the component c of claim 1 are at least one sort in the formation of a partial acrylic which is Component a, or a partial methacrylic-ized epoxy resin. Frame sealing-compound constituent of the liquid crystal equipment of claim 1 which is 20 - 80 weight section to the 100 weight sections.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] this invention relating to the frame sealing-compound constituent of a liquid crystal display, and setting the content of the water meltable ionicity matter to a detail whenever [below a predetermined value / ionic conduction] — especially, it is related with the frame sealing-compound constituent which can manufacture a high-definition liquid crystal display simply in a short time.

[0002]

[Description of the Prior Art] This kind of liquid crystal display is produced in recent years by the approach shown in drawing 1. In drawing 1 (a), 1 is one glass substrate of the glass substrates for liquid crystal of a pair with which an electrode pattern and the orientation film were given, and first, as shown in drawing 1 (b), it forms the frame sealing compound 2 on one glass substrate 1. At this time, it is lacking in the frame sealing compound 2 in this part, and the liquid crystal injected hole 3 is formed in it.

[0003] Subsequently, after making the glass substrate 4 of another side counter on the frame sealing compound 2 of a glass substrate 1 as while is shown in drawing 1 (c), the cel 5 for liquid crystal enclosure which was made to harden the frame sealing compound 2 with heating etc., and was surrounded in the glass substrates 1 and 4 of a pair by the glass substrates 1 and 4 and the frame sealing compound 2 of lamination and a pair is formed.

[0004] As subsequently to drawing 1 (d) shown in this cel 5, after pouring in liquid crystal 6 from the liquid crystal injected hole 3 in a vacuum, the liquid crystal injected hole 3 is sealed and a liquid crystal display 7 is manufactured.

[0005] As a frame sealing compound 2 of the above-mentioned liquid crystal display 7, the heat-curing mold adhesives which make an epoxy resin a subject are used conventionally. Hardening takes several hours at the elevated temperature before and behind 150 **, for this reason, the fall of the adhesion of the glass substrate by heat distortion, generating of a location gap of a fine pattern, and the variation of a gap arise, and this poses a big problem especially in production of a high-definition liquid crystal display.

[0006] Moreover, what uses together the photo-curing which uses the formation of a partial acrylic of a novolak mold epoxy resin or a partial methacrylic ghost as a principal component as shown in the acrylic adhesives of the photo-curing mold which uses acrylic ester or methacrylic ester as a principal component, the epoxy system adhesives of a photo-curing mold, or JP,3-188186,A, and heat curing is proposed.

[0007] Since a frame sealing compound hardens in ordinary temperature and a short time by the exposure of light in the case of these photo-curing mold adhesives and the glass substrate for liquid crystal is fixed promptly, the trouble at the time of using the above-mentioned epoxy resin is canceled. Or surely the advantage on the fabrication operation that production does not take long duration etc. exists.

[0008] However, the level it may fully be satisfied with the bottom of the adhesive property originally demanded as a property of a frame sealing compound and high-humidity/temperature of a level on the other hand in many property sides of not causing maintenance of the electro-optics property of the liquid crystal at the time of carrying out long duration neglect and orientation turbulence is not reached.

[0009] Furthermore, about the property that electrical-potential-difference retention does not fall at the time of the bottom long time neglect of high-humidity/temperature most important for the thin film transistor mold high definition liquid crystal display for the remarkable full color display of development recently, any adhesives for photo-curing shuttering seals run short efficiently, and you may say that the consideration of countermeasures to this is not made at all.

[0010]

[Problem(s) to be Solved by the Invention] Then, by using a constituent unique as a frame sealant, the purpose of this invention produces a high-definition liquid crystal display simply for a short time, may improve fabrication operation nature, and is to offer the frame sealing-compound constituent of the liquid crystal display which improved the fault which consists in the above-mentioned well-known technique at coincidence.

[0011]

[Means for Solving the Problem] In order to attain the above-mentioned purpose, according to this invention, the following components a-e, At least a bisphenol A mold epoxy resin, an acrylic acid, or a methacrylic acid among the formation of a partial acrylic reacted and obtained, or a partial methacrylic-ized epoxy resin Namely, a kind, b) Acrylic ester, methacrylic ester monomers, or these oligomer, c) It is the hardenability compound which uses an epoxy resin, d) photopolymerization initiator, and e) latency epoxy curing agent as an indispensable component, and is

characterized by the content of the water meltable ionicity matter being 40 or less second/cm in whenever [ionic conduction].

[0012]

[Detailed Description of the Invention] The formation of a partial acrylic or partial methacrylic-ized epoxy resin which is a resultant with above-mentioned a component, i.e., the bisphenol A mold epoxy resin and an acrylic acid, or a methacrylic acid is obtained as follows. First, the bisphenol A mold epoxy resin, an acrylic acid, or a methacrylic acid is reacted in 0.9-1.1Eq of carboxylic-acid radicals to 2Eq of epoxy groups under existence of a basic catalyst according to a conventional method.

[0013] Subsequently, after adding about 4 times as many toluene as this and the pure water of tales doses to this resultant by the weight ratio and stirring at 60-80 degrees C for 1 hour, it puts, and separates into an organic layer and a water layer, and a water layer is removed. This actuation is repeated 3 to 5 times, and the formation of a partial acrylic or partial methacrylic-ized epoxy resin which removed the toluene which finally collects organic layers and remains by vacuum distillation, and reduction-ization-processed the water meltable ionicity matter is refined.

[0014] as the example of the above-mentioned bisphenol A mold epoxy resin — Epicoat 850, and 860 and 4055 — [828, 834, 1001 and 1004 [the product made from oil-ized Shell Epoxy], and Epiclone () by Dainippon Ink & Chemicals, Inc. etc. is mentioned.) [for example,] the resin which performed reduction-ized processing (henceforth high grade-ized processing) of the water meltable ionicity matter preferably as these raw material resin, Epiclone 850S [for example,], — () by Dainippon Ink & Chemicals, Inc. etc. is suitable.)

[0015] The acrylic ester, the methacrylic ester monomer, or oligomer which is the above-mentioned component b is not limited to these, although 2-hydroxyethyl acrylate or methacrylate, JI, Tori, tetraethylene glycol diacrylate or methacrylate, bisphenol A denaturation diacrylate or methacrylate, urethane denaturation diacrylate, or methacrylate is specifically mentioned. Although these monomers or oligomer also performs high grade-ized processing if needed, this processing is usually performed by re-superfractionation.

[0016] As an epoxy resin of the above-mentioned component c, the bisphenol A mold epoxy resin, a bisphenol female mold epoxy resin, a novolak mold epoxy resin, a ring type aliphatic series epoxy resin, etc. are mentioned.

[0017] Considering the thing of the same kind mentioned to the above-mentioned component a about the bisphenol A mold epoxy resin as a bisphenol female mold epoxy resin, it is Epicoat. 807 [the product made from oil-ized Shell Epoxy] Epiclone 830 [the Dainippon Ink & Chemicals, Inc. make] etc. includes a phenol and cresol mold resin as a novolak mold epoxy resin, and it is Epicoat. There are 152, 154, Epiclone N-730, N-740, N-655, and N-670 grade. Moreover, there are commercial items, such as EOCN-1020, -1025, and -102 [the product made from Japanese Powder], in a high grade cresol novolak mold epoxy resin. As a ring type aliphatic series epoxy resin, ERL 4221, 4206, and 4229 [the product made from UCC] can be mentioned.

[0018] When commercial high grade resin cannot come to hand among these epoxy resins, high grade-ization may be performed by the usual molecular distillation method etc. Moreover, of course, the resin of a different type on the occasion of combination may be compounded and used.

[0019] Specifically, the photopolymerization initiator which is the above-mentioned component d can mention benzophenone, 2, and 2-diethoxy acetophenone, benzyl, benzoyl isopropyl ether, benzyl dimethyl ketal, 1-hydroxy cyclohexyl phenyl ketone, a thioxan ton, etc.

[0020] The latency epoxy curing agent which is the above-mentioned component e is a heat hardening mold, and it is more desirable than the standpoint which uses the last combination constituent as a 1 liquid type combination constituent preferably for low temperature and short-time hardening to be possible as much as possible.

[0021] Specifically as these latency curing agent, there are an imidazole derivative, NI organic-acid dihydrazide, etc. which are represented by aromatic amines, such as an I meta-phenylenediamine, diamino diphenylmethane, and diamino diphenyl sulfone, a RO dicyandiamide, Ha cure ZORU OR, cure ZORU CN, and cure ZORU AZINE (Shikoku Chemicals Corp. make).

[0022] Moreover, after dissolving and adding sufficient stirring, warming using either of the mixed solutions of ** methanol, ** methanol, the mixed solution of an organic solvent and ** methanol, and pure water as a high grade-ized art about these latency curing agents, the approach of repeating filtration actuation, drying and refining can be used.

[0023] In this invention, friend cure-VDH, -LDH, and -UDH[Ajinomoto Co., Inc.] is especially the most effective for special organic-acid dihydrazide and a concrete target among the above-mentioned latency curing agents.

[0024] In addition to the above-mentioned indispensable component of a-e, this invention may blend an inorganic bulking agent as a component f further. Specifically, a synthetic silica, talc, etc. can be mentioned as this inorganic bulking agent. Although high grade-ized processing is performed if needed also about this component, a desired refined material can be obtained by repeating washing using pure water as this approach.

[0025] Furthermore, this invention may blend the spacer for securing the coupling agent for improving the thixotropy agent which prepares the furniture of combination liquid in addition to each above-mentioned component, and an adhesive property, an additive, and a predetermined gap etc. However, it is indispensable that the content of the water meltable ionicity matter of the last combination constituent holds cm in 40 or less microseconds /by whenever [ionic conduction] on the occasion of combination of these components.

[0026] It combines and, specifically, said thixotropy agent is that vinylsilane, an epoxy silane, an amino silane, the mercapto silane of the silane coupling agent for a silicic anhydride and adhesive amelioration, etc. are independent, or the polymer bead with which the additive was adjusted to size predetermined mainly in the spacer of gap ***, such as denaturation oligomer of a butadiene-acrylic nitril copolymer.

[0027] The loadings of above-mentioned each component are the weight section, and are the a component 100. To the section, the 20 to 80 section, b component is the 30 to 70 section, and c component is the 30 to 70 section preferably the 20 to 80 section. Furthermore, d component of 0.5 - 3% of the weight of total weight and e component is the same, and other components are chosen for f component zero to 40% of the weight three to 25% of the weight by 1 - 5% of the weight of within the limits.

[0028] As for the hardenability compound constituted by the above-mentioned component, it is indispensable that the content of the water meltable ionicity matter holds 40 or less second/cm by whenever [ionic conduction] for example, among a hardened material, and it holds 15 or less second/cm preferably. Whenever [this ionic conduction] is measured using the conductivity meter for example, by Horiba, Ltd.

[0029] In addition, especially, an above-mentioned hardenability compound fully kneads an inorganic bulking agent etc. with careful attention to the homogeneity of a solid, and perfect distribution using a paint roll etc., and obtains the frame sealing-compound constituent of the liquid crystal display concerning this invention.

[0030] The frame sealing-compound constituent constituted as mentioned above is used for production of a liquid crystal display as shown in above-mentioned drawing 1. That is, in drawing 1, using the glass substrate 1 of drawing 1 (a), as shown in drawing 1 (b), the frame sealing compound 2 which starts this invention on a glass substrate is formed. Subsequently, the glass substrate 4 of another side is made to counter on the frame sealing compound 2 of a glass substrate 1, as while is shown in drawing 1 (c). 1000-3000mJ carries out the amount exposure of the beams of light, such as ultraviolet rays, under pressurization, and the frame sealing compound 2 is solidified. The glass substrates 1 and 4 of a pair Lamination, Further after that, while it has been non-energized, it heats at the temperature of 100 - 120 ** for about 1 hour, and it fully hardens, and the cel 5 for liquid crystal enclosure surrounded by the glass substrates 1 and 4 and the frame sealing compound 2 of a pair is formed. As subsequently to drawing 1 (d) shown in this cel 5, after pouring in liquid crystal 6 from the liquid crystal injected hole 3 in a vacuum, the liquid crystal injected hole 3 is sealed and a liquid crystal display 7 is manufactured.

[0031]

[Function] Since 40 or less second/cm of contents of the water meltable ionicity matter is 15 or less second/cm preferably in whenever [ionic conduction], the frame sealing compound which consists of an above-mentioned configuration obtains a high-definition liquid crystal display.

[0032] By having, first, by the photo-curing of a first stage story, a further above-mentioned frame sealing compound solidifies the whole frame sealing compound, fixes both the glass substrates of a liquid crystal display, and prevents a location gap of a glass substrate and the variation of a gap. Next, heat curing is performed on a second stage story. Since the frame sealing compound is already solidified on the first stage story at this time and the glass substrate is fully mutually fixed by cohesive force with high Component a, a process to which a liquid crystal substrate is fixed to a metal fixture, and a fixture carries out long duration heat hardening processing within a heating furnace like the conventional technique is unnecessary at all. That is, it is possible to only carry out heat hardening of the liquid crystal glass substrate after first stage story photo-curing termination in the state of a fixture free-lancer as it is, during heating, a glass substrate cannot separate, or gap change cannot be caused, and a good liquid crystal display can be produced. This means that online-izing is possible in the lamination process of a liquid crystal glass substrate, and brings about a big cost cut in manufacture of a liquid crystal display.

[0033] If the above-mentioned this invention operation is explained further in full detail, to fault points, such as location **** of the pattern first generated by heat hardening, and variation of GIYYAPU, the function of photo-curing is given through the acrylic component of the components a, b, and d in which room temperature setting is possible, and the glass substrate which faces by solidifying the whole frame sealing compound by optical exposure on the first stage story of hardening will be fixed, and it will be coped with. Next, through the epoxy system component of Components a, c, and e, the manifestation of the firm adhesive strength to a glass substrate and the environmental-test strength over a long period of time constitutes the structure of cross linkage effective in a system by the heat hardening of a second stage story, and copes with it.

[0034] furthermore, generally an important thing has the acrylic component and the epoxy component corresponding to heat hardening corresponding to photo-curing during the same combination presentation, in the case of a process in which especially hardening passes through gradual hardening with light and heat, may be boiled occasionally, may be done, and the whole resin may present demixing and a layer separation phenomenon In this case, a result which weakens the cohesive force of adhesives remarkably is brought, and it becomes a very serious fault. Component a is blended as a component corresponding to this trouble, and since Component a is the configuration of sharing an acrylic radical and an epoxy group in the same molecule, like the above-mentioned, the above-mentioned layer separation phenomenon is completely solvable by blending this into a system with the compatibility-ized capacity over b component peculiar to a component, and c component. Since a component reacts to coincidence with b acrylic monomer or oligomer through the acrylic radical, and an epoxy group is minded, and it reacts and combines with an epoxy resin, a component which has an acrylic radical and an epoxy group in the same molecule will constitute the bridge formation network which attains to the whole system positively, and, therefore, will contribute to the improvement in the engine performance about bond strength, thermal resistance, and an elevated temperature and moisture resistance greatly.

[0035] As mentioned above, since it consists in the above views, it is an indispensable indispensable combination component to surely blend the component c used as the component b used as the component a which has two sorts in this invention of different reaction radicals in the same intramolecular, and acrylic resin, and an epoxy resin during the same combination presentation. And by blending the blending ratio of coal of the combination components

a, b, and c to the component a 100 weight section within the limits of the 20 to b80 section, and the 20 to c80 section, each resinous principle hardens completely in each phase of photo-curing and heat curing, and the engine performance suitable as a frame sealing compound is discovered. It is the result of finding out that selection of the molecule which has moderate chain length is that to which the long chain length gives good flexibility easily to the whole hardened material, after that versatility experiments to have used the bisphenol A mold epoxy resin as an epoxy resin of Component a.

[0036] In this invention, the further most important thing is that the original purpose of this invention is in offer of the frame sealing compound for high-definition liquid crystal displays, therefore it being the need are not causing at all the orientation turbulence of the liquid crystal with which a frame sealing compound's becomes a cause especially to the environmental test of long duration in addition to aforementioned all items, and maintaining electrical-potential-difference maintenance with a high level. for that purpose, the thing for which a frame sealing compound does not have the water meltable ionicity matter as much as possible in the system, if it puts in another way It is indispensable absolutely that it is a high grade very much. Also about the purity level The water meltable ionicity matter of a hardened material is extracted on certain conditions about the hardened material of this combination constituent using ultrapure water as a result of an experiment and examination. keeping desirable 40 or less second/cm of this at 15 or less second/cm paying attention to whenever [ionic conduction] — the need — enough — this invention persons found out that they were conditions.

[0037] This invention repeats examination wholeheartedly also about the approach of this high-grade-izing, and devises and enforces the most suitable high grade-ized art about each raw material component again.

[0038]

[Example] Hereafter, an example is given and this invention is explained concretely. First, the formation of bisphenol A mold epoxy resin partial methacrylic one or acrylic ghost which is Component a was compounded by the approach as shown in the following synthetic example.

[0039]

[A synthetic example]

b) high grade bisphenol A mold epoxy resin: —] by Epiclone-850S[Dainippon Ink & Chemicals, Inc. — the 1000 weight sections (the following, the section, and display), the methacrylic-acid:250 section, the toluene:900 section, the triethylamine:2 section, and PARAME — an ibis — the CIF enol:2 section was mixed, heating stirring was carried out at 90 degrees C for 8 hours, and the partial addition reaction object was obtained.

[0040] b) The toluene:4500 section is added to the product of above-mentioned I, and consider as diluted solution, put after adding the pure-water:4500 section to this and stirring at a room temperature for 1 hour, and separate and remove a water layer. 3 to 5 times, washing according washing according this washing actuation to the 1 convention NaOH solution of tales doses only to the pure water of tales doses was further repeated 3 to 5 times 3 to 5 times to the degree, whenever [that ionic conduction] was measured about the last wash water using : conductivity meter [by measuring instrument [Horiba, Ltd.] whenever [ionic conduction], and it checked that they were 10 or less second/cm.

[0041] c) The solution which filtered and obtained the solution of above-mentioned RO was condensed at 70 degrees C under reduced pressure, full removal purification of the toluene was carried out, and the partial methacrylic-ized epoxy resin was compounded.

[0042] Moreover, according to said synthetic example, it changed to the methacrylic acid and the partial acrylic-ized epoxy resin was compounded by the same approach using the acrylic acid.

[0043] Next, high grade-ized processing about each component was carried out as follows.

Example [of high grade-ized processing] a component: It carried out by the approach of the above-mentioned synthetic example.

b) Component : about acrylic ester or a methacrylic ester monomer, and oligomer, when it was a hypoviscosity solution, it distilled and refined by the approach by high vacuum distillation, and, in the case of the hyperviscous solution, the dilution and the washing approach using the same toluene and pure water as a high grade-ized art of the above-mentioned a component were repeated and enforced.

c) Component : about the epoxy resin, the high grade-ized epoxy resin of a commercial item was used as it was.

d) Component : about the photopolymerization initiator, after the superfluous methanol washed the solid 3 to 5 times, it was dried and used with the oven.

e) component: — a latency epoxy curing agent — the solid — a superfluous methanol — using — once — warming — it dissolved, and after stirring, the actuation which filters, cools, is crystallized and is collected was repeated 3 to 5 times, and was carried out.

f) Component : about the inorganic bulking agent, after repeating the actuation which uses superfluous pure water, and is stirred and washed 3 to 5 times, it was dried and used with the oven.

[0044] By the above all directions method, although high grade-ized processing was performed about each component, it was used by performing high grade-ization until it measured whenever [in the wash water of the last / ionic conduction] and became 10 or less second/cm preferably cm 30 or less microseconds /about the approach of washing especially using pure water.

[0045]

[Example 1]

a) component: — partial methacrylic-ized epoxy resin: produced in said synthetic example — the 30 sections b) — component: — I JISHIKURO cutting-pliers nil acrylate: — the six sections and RO bisphenol A dimethacrylate: —

the 6 sections c) — component: — the S:Epicon 850 15 section and d component: 1-hydroxy cyclohexyl phenyl ketone: — the 1.5 sections e) — component: — the friend cure-VDH:14 section and f component: —]made from S quartz watch M-2010[Nippon Steel Chemistry: — the 25 sections KBM-403[silane coupling agent: The][by Shin-Etsu Chemical Co., Ltd.]:2.5 section was fully kneaded using the paint roll, and the frame sealing compound (A) of about 100,000 centipoises viscosity was produced.

[0046] Moreover, the purity of this frame sealing compound (A) was measured by the following approach, and was checked. The UV irradiation machine which makes a high-pressure mercury lamp the light source is used, and they are 100 mw/cm². It irradiates for 30 seconds, hardens and is in oven about this hardened material further. It heated for 120 degree-Cx 1 hour, and full hardening was carried out. About 1g of this hardened material is taken, frost shattering is cooled and carried out by liquid nitrogen, and it is ultrapure water abbreviation to that detailed powder. 100g is added, pressure cooker equipment is used, and it is under pressurization. It put at 121 degrees C for 24 hours, and the water meltable ionicity matter was extracted. When whenever [ionic conduction / of this extract water] was measured, it is 8.9 second/cm and has checked that it was a desired high grade object.

[0047]

[Example 2] In the combination presentation of an example 1, the partial methacrylic-ized epoxy resin of a component was changed into the partial acrylic-ized epoxy resin of said synthetic example, and, similarly other components and compounding ratios produced the frame sealing compound (B) of about 100,000 centipoises viscosity. Moreover, it has checked that whenever [ionic conduction / of this thing] was 11.5 second/cm, and was a desired high grade object similarly.

[0048]

[Example 3] In the combination presentation of an example 1, with the combination constituent except the acrylic or the methacrylic monomer, and oligomer of b component, the rate of a compounding ratio was made into the a component:40 section, the c component:22 section, the d component:1 section, the e component:20 section, the f component:15 section, and the KBM-403:2 section, the same actuation as an example 1 was performed, and the frame sealing compound (C) of about 300,000 centipoises viscosity was produced.

[0049]

[Example 4] In the combination presentation of an example 1, with the combination constituent except the epoxy resin of c component The rate of a compounding ratio was made into the a component:40 section, the b component:10 section, the RO 16 section, the d component:2 section, the e component:5 section, the f component:25 section, and the KBM-403:2 section, the same actuation as an example 1 was performed, and the frame sealing compound (D) of about 40,000 centipoises viscosity was produced.

[0050]

[The example 1 of a comparison] Commercial ultraviolet-curing mold adhesives: The world lock 815 [the Kyoritsu Chemical & Co., Ltd. make] was used as a frame sealing compound as it was. Moreover, whenever [ionic conduction / of this frame sealing compound] was 75 second/cm.

[0051] Here, the bond strength to glass was first measured [1st] about each frame sealing compound shown in the aforementioned example. The ultralow volume of a frame sealing compound was taken on slide glass, it pushed so that it might be in the core of the glass with which superposition and these adhesives laid other slide glass on top of the cross-joint mold on it and adhesives thickness might become 8-10 micrometers and the diameter of about 3mm, and the piece of an adhesion test was produced. They are 100 mw/cm² to this test piece. Non-energized as it is, after it irradiates UV light for 30 seconds and it carries out photo-curing Heat hardening was carried out for 60 minutes in 120-degree C oven, and the bond strength measurement test piece was produced.

[0052] The following result was obtained when bond strength was measured about this test piece using the tension gauge.

a) Frame sealing-compound [of an example 1] (A) — 1.50 Frame sealing-compound [of the kg/mmb example 2] (B) — 1.25 sealing-compound [of the kg/mmc example 3 / frame] (C) — 0.08 sealing-compound [of the kg/mmd example 4. / frame] (D) — The frame sealing compound of the example 1 of a 0.25 kg/mme comparison — It is shown that the test result of the 0.78 kg/mm above is excellent in a frame sealing compound (A) and (B) in bond strength compared with other frame sealing compounds.

[0053] Next, the cel for liquid crystal displays was produced to the 2nd, and the existence of orientation turbulence was examined. To the frame sealing compound of the examples 1-4 acquired above and the example 1 of a comparison, it is a diameter. Spacer with a 6.5-micrometer die length of 1mm Mix the 0.5 sections and a predetermined pattern is printed on the glass substrate 1 of drawing 1 using a screen printer. The frame sealing compound 2 is formed and, subsequently it is drawing 1 (c). After making the glass substrate 4 of another side counter like, Using the pressurization ultraviolet curing equipment-light press for lamination [the product made from Run Technical service], it pressurizes at a room temperature for 1 to 3 minutes, and is gap thickness. 6.5 micrometers They are 100 mw/cm² in the condition of having carried out. UV light was irradiated for 30 seconds, and carried out photo-curing, and the cel 5 for liquid crystal enclosure was produced. 3 is a liquid crystal injected hole.

[0054] Next, this cel 5 was taken out from the optical press machine, and the cel 5 which added and carried out full hardening of the 1-hour heating in the oven of 120 ** as it was (a fixture is not attached) was produced. In addition, in the aforementioned production approach, heat hardening did not carry out about the frame sealing compound of the example 1 of a comparison. As shown in the cel 5 for liquid crystal enclosure obtained as mentioned above in a vacuum at drawing 1 (d), from the liquid crystal injected hole 5, liquid crystal 6 was poured in and the liquid crystal

display 7 was produced.

[0055] About each of this liquid crystal display, it is under the conditions of 65 degrees C and 95%RH. The elevated temperature and the highly humid performance test of 500 hours were performed, the existence of orientation turbulence was investigated, and the following result was obtained.

配向乱れ

- a) 実施例 1 の枠シール剤 (A) ... 無 し
- b) 実施例 2 の枠シール剤 (B) ... 無 し
- c) 実施例 3 の枠シール剤 (C) ... 有 り
- d) 実施例 4 の枠シール剤 (D) ... 有 り
- e) 比較例 1 の枠シール剤 ... 有 り

A frame sealing compound (A) and (B) do not cause orientation turbulence, but the above-mentioned test result shows the thing of a significant difference to be clearly.

[0056] Next, the cel for TFT (thin film transistor) liquid crystal displays is produced by the same approach as the above to the 3rd, and it is under the conditions of 65 degrees C and 95%RH. The elevated temperature and the highly humid performance test of 500 hours were performed, and the electrical-potential-difference retention after a trial was examined. For the result of a trial, the frame sealing compound was as follows using the frame sealing compound of an example 1 and the example 1 of a comparison.

電圧保持率

- a) 実施例 1 の枠シール剤 (A) ... 99.2 %
- b) 比較例 1 の枠シール剤 ... 63.4 %

Having effect remarkable [the above-mentioned test result], the content, i.e., the difference of purity, of the water meltable ionicity matter with a frame sealing compound (A) and the frame sealing compound of the example of a comparison, in electrical-potential-difference retention is shown.

[0057]

[Effect of the Invention] As explained in full detail above, the frame sealing compound of this invention has sufficient bond strength to a glass substrate, does not cause orientation turbulence in a practical use side, but especially, has high electrical-potential-difference retention also in the high-definition TFT mold liquid crystal display, and shows display property sufficient as a combination constituent of a frame sealing compound, and environmental endurance. Moreover, also in the manufacture side of a liquid crystal display, improvement and a cost cut of remarkable workability can be aimed at from the ability of the hardening approach by the pressing fixture free-lancer at the time of short-time hardening by photo-curing, and heat hardening to be adopted, and it is practically very useful.

[Translation done.]